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(54) **LUBRICATING AGENT FOR MOLD AT ELEVATED TEMPERATURE, IRON-BASED POWDER COMPOSITION FOR ELEVATED TEMPERATURE COMPACTION WITH LUBRICATED MOLD AND HIGH DENSITY FORMED PRODUCT FROM IRON-BASED POWDER COMPOSITION, AND METHOD FOR PRODUCING HIGH DENSITY IRON-BASED SINTERED COMPACT**

(57) A process for producing a high-density iron-based green compact is provided which can form a green compact with a high density. Also provided is a process for producing a sintered compact from the green compact. A specified combination lubricant is applied to the surface of a die for pressure compaction by electrical charging, which lubricant is composed of a lubricant having a higher melting point than a preset compaction temperature, and a lubricant having a lower melting point than the compaction temperature. A heated iron-based powder mixture is filled into the die, fol-

lowed by pressure compaction, whereby a green compact is formed. The green compact can be sintered to provide a sintered compact. The powder mixture comprises an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] This invention relates to processes for the production of iron-based powder green compacts and iron-based sintered compacts for powder metallurgy. More particularly, the invention relates to improvements in lubricants for use in producing a high-density, green compact made from iron-based powder by warm compaction.

2. Description of the Related Art

[0002] In general, a powdered iron-based green compact for powder metallurgy is produced by filling an iron-based powder mixture into a die. The powder mixture is generally derived by mixing an iron-based powder with alloying powders such as copper powder, graphite powder and the like and further with lubricants such as zinc stearate, lead stearate and the like, and then by subjecting the iron-based powder mixture to. The resultant green compact usually has a density in the range from 6.6 to 7.1 Mg/m³.

[0003] Such a green compact is further sintered to obtain a sintered compact which, where desired, is sized or cut into a powder metallurgical product. Where great strength is required, carburizing heat treatment or brightening heat treatment is in some instances performed after completion of the sintering.

[0004] The above powder metallurgy permits components parts of complicated shapes to be formed with high dimensional accuracy and in near net structure, significantly saving the cost of cutting work as contrasted to conventional production methods.

[0005] With regard to powder metallurgical iron products, a keen demand has recently been made for more higher dimensional accuracy to omit cutting work and to save production cost, and also for more greater strength to make components parts small in size and light in weight.

[0006] In order to give greater strength to a powder metallurgical product (a sintered compact), it is beneficial to form high-density sintered compacts from an iron-based green compact which has been produced to have a high density. As the density of a sintered compact increases, the number of voids in the compact decreases so that the component part is obtainable with improved mechanical properties such as tensile strength, impact value, fatigue strength and the like.

[0007] As warm compaction techniques evolved to form a high-density iron-based green compact, there have been proposed a double molding-double sintering method in which an iron-based powder mixture is pressed and sintered in the usual manner, followed by repeated pressing and sintering, and a sinter forging method in which single pressing and single sintering are performed, followed by hot forging.

[0008] Moreover, warm compaction techniques are known in which metal powder is compacted with heat as disclosed for instance in Japanese Unexamined Patent Application Publication No. 2-156002, Japanese Examined Patent Application Publication No. 7-103404, U.S. Patent No. 5,256,185 and U.S. Patent No. 5,368,630. Such a warm compaction techniques are designed to melt and disperse a lubricant partly or wholly between the metallic particles, thereby reducing the frictional resistance between the metallic particles and the frictional resistance between the green compact and an associated die, so that improved compressibility is attained. The compaction technique noted here is thought to be most advantageous in view of possible cost savings amongst the methods previously mentioned for the production of high-density iron-based green compacts. A green compact of about 7.30 mg/m³ in density can be obtained by the above compaction technique when an iron-based powder mixture is compacted at a pressure of 7 t/cm² and at a temperature of 150 C, which powder mixture is derived by mixing a partially alloyed iron powder of a Fe-4Ni-0.5Mo-1.5Cu with 0.5% by mass of graphite and 0.6% by mass of lubricant.

[0009] However, according to the warm compaction techniques of the above-cited publications, i.e., Japanese Unexamined Patent Application Publication No. 2-156002, Japanese Examined Patent Application Publication No. 7-103404, U.S. Patent No. 5,256,185 and U.S. Patent No. 5,368,630, the problem arises that an iron-based powder mixture is less fluid and hence less productive, the resultant green compact is irregular in respect of densities, and the resultant sintered compact is unfavorably variable in respect of physical properties. Another drawback is that a high force must be applied to draw the green compact from the corresponding mold with consequent marred surface of the product and shortened lifetime of the die.

[0010] In these warm compaction techniques, a lubricant is also contained in an iron-based powder mixture so as to reduce the resistance between the metallic particles and the resistance between the green compact and the associated mold, thereby providing improved compressibility. During warm compaction, the lubricant is partly or wholly melted and then pushed to locate adjacent to the surface of the green compact. Upon subsequent sintering, the lubricant gets thermally decomposed or volatilized and hence escapes from the green compact, leaving coarse voids near to the

surface of the sintered compact. This poses the problem that the sintered compact results in insufficient mechanical strength.

[0011] To cope with this problem, Japanese Unexamined Patent Application Publication No. 8-100203 discloses that when room temperature compaction or warm compaction is effected, the content of a lubricant may be incorporated in an iron-based powder mixture is decreased by coating the surface of a die with an electrical charged lubricant powder such that a high-density green compact is produced. In this technique, however, the coating lubricant is susceptible to morphological changes at around its melting point since it is of a single nature so that the lubricating action is largely variable. This has the drawback that the compaction temperature range depends restrictedly upon the melting point of the coating lubricant. Also defectively, even if the content of the lubricant in the powder mixture can be decreased with the coating lubricant applied on to the mold surface, the content of the former lubricant may be too low which is dependent upon the lubricant components to be incorporated in the powder mixture. In this instance, the former lubricant does not exhibit lubrication, failing to enhance the density of a pressurized powder.

[0012] From the viewpoints of great strength and cost saving of automotive parts, there has been a need for the development of a process capable of producing an iron-based green compact with a higher density but by single compaction.

SUMMARY OF THE INVENTION

[0013] In order to eliminate the foregoing problems of the conventional art, a first object of the present invention is to provide a process for producing a high-density iron-based green compact which permits a high-density green compact to be formed with a density of 7.4 Mg/m^3 or above and by single pressing when warm pressure compaction is effected as to an iron-based powder mixture derived by mixing a partially alloyed iron powder of, for example, a Fe-4Ni-0.5Mo-1.5Cu composition, with 0.5% by mass of a graphite powder.

[0014] A second object of the invention is to provide a process for producing a high-density iron-based sintered compact which permits a high-density sintered compact to be formed by sintering such an iron-based green compact.

[0015] To achieve the above objects by utilizing a warm compaction technique and a die lubrication compaction technique, the present inventors have conducted intensive researches on various lubricants for mold lubrication and various formulations of iron-based powder mixtures containing lubricants. It has now been found that the force for drawing an iron-based green compact from the corresponding mold can be effectively lessened by the use of a certain specific combination lubricant as a lubricant for mold lubrication. This combination lubricant is composed in a suitable ratio of a lubricant having a lower melting point than a preset compaction temperature and a lubricant having a higher melting point than the compaction temperature and can be applied to the surface of a preheated die by electrical charging.

[0016] The present invention has been made on the basis of the aforesaid finding and further supporting studies.

[0017] More specifically, according to a first aspect of the present invention, there is provided a lubricant for warm mold lubrication, comprising a mixture of a lubricant having a higher melting point than a preset compaction temperature, and a lubricant having a lower melting point than the compaction temperature, the lubricant for mold lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted in the mold by pressure compaction.

[0018] According to a this invention, there is provided a die lubricant for warm compaction with die, comprising a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance, the lubricant being applicable to a surface of a preheated die by means of electrical charging when a powdered material is compacted in the mold by pressure compaction.

[0019] In this aspect, the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.

[0020] In this aspect, the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

[0021] According to a second aspect of the invention, there is provided an iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder and a powder compaction lubricant, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

[0022] According to this aspect of the invention, there is provided an iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the

graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0023] In the second invention, the content of the powder compaction lubricant is in the range from 0.05 to 0.40% by mass.

[0024] According to the third invention, there is provided a process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a die lubricant for warm compaction with die to the surface of the mold by electrical charging; filling a heated iron-based powder mixture in the mold; and then subjecting the mixture to pressure compaction at a preset compaction temperature, wherein the die lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder and a powder compaction lubricant, the powder compaction lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

[0025] In this third invention, the graphite powder can be also added in a content less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0026] In the third invention, the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a layer crystal structure.

[0027] The lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

[0028] The lubricant for in the powder mixture is preferably added in a amount from 0.05 to 0.40% by mass.

[0029] The present invention can also provide a high-density sintered compact produced by single pressing.

[0030] In a fourth embodiment of the invention, there is provided a process for the production of a high-density iron-based sintered compact, comprising the step of further sintering the high-density iron-based green compact produced by the process according to any one of the fifth and sixth aspects, thereby forming a sintered compact.

[0031] The above and other objects, features and advantages of the present invention will become manifest upon reading of the following detailed description.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0032] In the practice of the present invention, a heated iron-based powder mixture is filled in a die and then molded by pressure compaction at a preset compaction temperature, whereby an iron-green compact is obtained.

[0033] In the invention, a die to be used is preheated at a suitable temperature. The preheating temperature is not particularly restricted so long as an iron-based powder mixture can be maintained at a preset compaction temperature. The preheating temperature is set to be preferably higher than the compaction temperature by 20 to 60 C.

[0034] An electrically charged lubricant for mold lubrication is introduced into a preheated die to apply the lubricant to the surface of the mold by electrical charging. Desirably, the lubricant (solid powder) for mold lubrication is placed in a die lubricating system (for example, Die Wall Lubricant System manufactured by Gasbarre Co.) where electrical charging is performed by means of contact charging between the solid lubricant particles and the inner wall of the system. The electrically charged lubricant is jetted into the mold and applied to the mold surface by electrical charging. The amount of the lubricant to be applied to the mold surface by electrical charging is set preferably in the range from 5 to 100 g/m². Amounts less than 5 g/m² result in insufficient lubricating action, calling for a high force to draw the resultant green compact from the mold. Amounts more than 100 g/m² cause the lubricant to remain on the product surface, making the product unsightly in appearance.

[0035] The die lubricant for warm compaction with die lubrication is used in electrically charged relation to the surface of a preheated die when a powdered material is compacted by pressure compaction. This lubricant is a mixture of a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance. The preset compaction temperature used herein denotes a temperature as measured on the mold surface at the time pressure compaction is carried out.

[0036] The higher-melting lubricant is present in a solid state in the die lubricant for warm compaction with die lubrication at the time compaction is effected, and it behaves like a solid lubricant that acts as "a roller" within a die, consequently lessening the force for drawing a green compact from the mold. Moreover, such higher-melting lubricant has a role to prevent a completely or partially molten lubricant (a lower-melting lubricant to be described later) from getting migrated within the mold, decreasing the frictional resistance between the green compact and the mold surface so that the force for product drawing is prevented from being unfavorably increased.

[0037] If the content of the higher-melting lubricant is less than 0.5% by mass, the lower-melting lubricant becomes

relatively abundant. This causes a large amount of a molten lubricant that migrates within a die and does not distribute uniformly on the surface of the mold, increasing the frictional resistance between the green compact and the mold surface and hence failing to lessen the force for product drawing to a sufficient extent. Conversely, if the content of the higher-melting lubricant is more than 80% by mass, a lubricant not subject to melting in a die is too large in amount for uniform distribution on the die surface. This is responsible for diminished mold lubrication and hence for increased force for product drawing. Hence, the content of the higher-melting lubricant present in the die lubricant for warm compaction with die lubrication should be within the range from 0.5 to 80% by mass.

[0038] The lubricant for mold lubrication contains, in addition to the above-specified higher-melting lubricant, a lubricant having a lower melting point than the preset compaction temperature. This lower-melting lubricant melts completely or partially at the compaction temperature and presents a grease-like state on the surface of a die, exerting a beneficial effect on lessening the force for drawing a green compact from the mold.

[0039] The higher-melting lubricant is preferably at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used:

[0040] As the metallic soap, zinc stearate, lithium stearate, lithium hydroxystearate or the like is preferred. As the thermoplastic resin, polystyrene, polyamide, fluorine resin or the like is preferred. As the thermoplastic elastomer, polystyrene elastomer, polyamide elastomer or the like is preferred. The inorganic lubricant of a lamellar crystal structure is graphite, MoS_2 or carbon fluoride, and finer particle sizes are more effective in lessening the force for product drawing. The organic lubricant of a lamellar crystal structure is melamine-cyanuric acid adduct (MCA) or N-alkyl aspartate-alkyl ester.

[0041] Meanwhile, the lower-melting lubricant is desired to be a lubricant that melts completely or partially at the compaction temperature and tends to be applied to the surface of a die at a low melting point by electrical charging. This lower-melting lubricant is preferably at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used. As the metallic soap, zinc stearate or calcium stearate is preferred. As the amide wax, ethylene bis-stearoamide, monoamide stearate or the like is preferred. As the eutectic mixture, ethylene bis-stearoamide-polyethylene eutectic, ethylene bis-stearoamide-zinc stearate eutectic, ethylene bis-stearoamide-calcium stearate eutectic is preferred.

[0042] Subsequently, a heated iron-based powder mixture is charged into a die electrically charged with a lubricant for mold lubrication, followed by pressure compaction, whereby a green compact is obtained.

[0043] The iron-based powder mixture is heated preferably at from 70 to 200 C. Lower temperatures than 70 C result in an iron powder having increased yield stress and hence lead to a green compact having decreased density. Inversely, higher temperatures than 200 C show no appreciable rise in density, arousing a fear of an iron powder getting oxidized. Thus, the temperature at which the iron-based powder mixture is heated should be set within the range from 70 to 200 C.

[0044] The iron-based powder mixture is derived by mixing an iron-based powder with a lubricant (a powder compaction lubricant) or an alloying powder. The method of mixing the iron-based powder with the compaction lubricant or the alloying powder is not particularly restrictive, but any known method is suitably useful. In the case where the iron-based powder is mixed with the alloying powder, it is desired that after completion of primary mixing in which the iron-based powder and alloying powder are mixed with a part of the powder compaction lubricant, secondary mixing be performed in which the resultant mixture is stirred with heat at a higher temperature than the melting point of at least one of the aforesaid lubricant in order to melt the one lubricant, and the mixture having been melted is cooled with stirring to thereby apply the one lubricant to the surface of the iron-based powder mixture so that the alloying powder is bonded, followed by mixing of the balance of the powder compaction lubricant.

[0045] The iron-based powder according to the present invention is selected from among pure iron powders such as an atomized iron powder, a reduced iron powder or the like, a partially diffusively alloyed steel powder, a completely alloyed steel powder, and a mixed powder thereof.

[0046] The content of the powder compaction lubricant in the iron-based powder mixture is set preferably in the range from 0.05 to 0.40% by mass based on the total amount of the iron-based powder mixture. Contents less than 0.05% by mass make the resultant iron-based powder mixture less fluid and fail to apply the lubricant uniformly to the surface of a die, producing a green compact having decreased density. Conversely, contents more than 0.40% by mass suffer high voiding after sintering and give a sintered compact having decreased density.

[0047] The powder compaction lubricant contained in the iron-based powder mixture is a mixed lubricant obtained by mixing a lubricant having a lower melting point than the preset compaction temperature and a lubricant having a higher melting point than the compaction temperature. The content of the lower-melting lubricant in the powder compaction lubricant is in the range from 10 to 75% by mass, whereas the content of the higher-melting lubricant is in the range from 25 to 90% by mass as the balance. The lower-melting lubricant is effective in that it melts during pressure compaction, penetrates in between the iron-based particles by capillary action, disperses uniformly in the particles, reduces particle-to-particle contact resistance and facilitates reorientation of iron-based particles, thus accelerating

the enhancement of product density. If the content of the lower-melting lubricant is less than 10% by mass, such lubricant fails to disperse uniformly in the iron-based particles and suffers poor density of a green compact. If the content of the lower-melting lubricant is more than 75% by mass, a molten lubricant is squeezed toward the surface of a die as the density of a green compact is increased so that passages are provided on the product surface for the molten lubricant to escape out of the product. The passages cause many coarse voids on the product surface, giving insufficient strength to the resultant sintered compact.

[0048] The higher-melting lubricant contained in the iron-based powder mixture is present in a solid state at the time compaction is effected. This lubricant acts as "a roller" on the surface protrusions of iron-based particles where it repels a molten lubricant, promoting particle reorientation and enhancing product density.

[0049] The higher-melting lubricant contained in the powder compaction lubricant for the iron-based powder mixture is preferably at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0050] As the metallic soap, zinc stearate, lithium stearate, lithium hydroxystearate or the like is preferred. As the thermoplastic resin, polystyrene, polyamide, fluorine resin or the like is preferred. As the thermoplastic elastomer, polyethylene elastomer, polyamide elastomer or the like is preferred. As the inorganic lubricant of a lamellar crystal structure, graphite, MoS_2 or carbon fluoride is preferred, and finer particle sizes are more effective for lessening the force for drawing a green compact from a die. As the organic lubricant of a lamellar crystal structure, melamine-cyanuric acid adduct (MCA) or N-alkyl aspartate-alkyl ester is preferred.

[0051] The lower-melting lubricant contained in the powder compaction lubricant for the iron-based powder mixture is preferably at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0052] As the metallic soap, zinc stearate, calcium stearate or the like is preferred. As the amide wax, ethylene bis-stearoamide, monoamide stearate or the like is preferred. As the eutectic mixture, ethylene bis-stearoamide-polyethylene eutectic, ethylene bis-stearoamide-zinc stearate eutectic, ethylene bis-stearoamide-calcium stearate eutectic or the like is preferred. Though dependent upon the compaction temperature used, some of these lower-melting lubricants may be utilized as higher-melting lubricants.

[0053] Graphite can be used as an alloying powder in the iron-based powder mixture. This graphite powder is effective to reinforce a sintered compact to be produced, but too high a content is liable to decrease product density largely. Hence, the content of graphite should be set to be less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0054] In the present invention, the high-density iron-based green compact formed by the above-specified production process can be further sintered, whereby a high-density iron-based sintered compact is obtained. Here, any conventional sintering method may be suitably used without limitation. Sinter hardening may also be used by which rapid cooling is effected after sintering to enhance product strength.

[0055] The present invention may be more fully understood with reference to the following examples.

Example 1

[0056] A partially alloyed steel powder of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder was used as an iron-based powder. Iron-based powder mixtures were prepared by mixing this alloyed steel powder with 0.5% by mass of a graphite powder and various lubricants shown in Table 1. The mixing was effected with heat and by use of a high-speed mixer.

[0057] First, a die for compacting was preheated at each of the temperatures listed in Table 1. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the mold die surface by means of electrical charging. The die lubricant for was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 1. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0058] Subsequently, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 1. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 1.

[0059] As a conventional example, a similar rectangular green compact (Green compact No. 38) was formed in the same manner as in Example 1 except that a die was not coated with a die lubricant for warm compaction with die.

[0060] After completion of the compaction, the force was measured which was required for the green compact to be

drawn from the mold.

[0061] With regard to each green compact thus formed, the density was determined by Archimedes' principle. The principle noted here denotes a method by which the density of a test specimen, each green compact in this case, is determined by measuring the volume of the product after immersion in ethyl alcohol. Additionally, visual inspection was made of the appearance of the green compact to find faults such as marring, breakage and the like. The green compact was centrally cut, embedded in resin and then abraded, followed by examination of voiding in section on a light microscope.

[0062] The drawing force, density, appearance and sectional structure of the green compact are tabulated in Table 1.

[0063] All the green compacts representing this invention show as low a drawing force after compaction as 20 MPa or below and as high a density as 7.4 Mg/m³ or above. Furthermore, these products are free of surface oxidation due to heating as well as faults such as marring, breakage and the like. The sectional structures are normal with the absence of coarse voids.

[0064] The comparative and conventional examples that fall outside the scope of the invention revealed a high drawing force exceeding 20 MPa, a low density of less than 7.35 Mg/m³, or coarse voids near to the sectional surface of the green compact.

[0065] Advantageously, the present invention can form a high-density green compact which exhibits superior appearance and sectional structure and low drawing force.

Example 2

[0066] The following six different powders were used as iron-based powders; namely (1) a partially alloyed steel powder a of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder, (2) a partially alloyed steel powder b of a Fe-2Ni-1Mo composition derived by diffusively bonding Ni and Mo to a pure atomized iron powder, (3) a prealloyed steel powder c of a Fe-3Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V, (4) a prealloyed steel powder d of a Fe-1Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V, (5) an atomized iron powder e, and (6) a reduced iron powder f. The atomized iron powder denotes an iron-based powder resulting from atomization of molten steel with high-pressure water, and the reduced iron powder denotes an iron-based powder resulting from reduction of iron oxide.

[0067] The partially alloyed steel powder a, partially alloyed steel powder b, prealloyed steel powder c, prealloyed steel powder d, atomized iron powder e and reduced iron powder f were each mixed with graphite in the contents shown in Table 3 and with the lubricants shown in Table 3, whereby iron-based powder mixtures were prepared. The mixing was effected with heat and by use of a high-speed mixer. In case of the atomized iron powder e and reduced iron powder f, 0.8% by mass of graphite and 2.0% by mass of a Cu powder were mixed. The content of graphite is by a mass ratio relative to the total amount of iron-based powder and graphite, or of iron-based powder, graphite and alloy powder.

[0068] First, a pressure compaction die was preheated at each of the temperatures listed in Table 3. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the mold and applied to the mold surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 3. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0069] Secondly, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 × 10 × 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 3. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 3.

[0070] With regard to each iron-based green compact thus obtained, the density was determined by Archimedes' principle as in Example 1.

[0071] Subsequently, the iron-based green compact was sintered in a N₂-10%H₂ atmosphere and at 1,130 C for 20 minutes, whereby an iron-based sintered compact was formed. The density of the sintered compact was determined by Archimedes' principle. This product was then machined to obtain a sample in the shape of a small round rod dimensioned to be 5 mm in parallel plane diameter and 15 mm in length. The sample used to measure tensile strength.

[0072] Similar rectangular green compacts were formed in the same manner as in Example 2 except that a die was not coated with a die lubricant for warm compaction with die. Each green compact was further sintered as in Example 2 to form an iron-based sintered compact which was taken as a conventional example.

[0073] The test results are tabulated in Table 3.

[0074] The present invention provides high density and great tensile strength in contrast to the conventional examples (Sintered compacts Nos. 2 to 12).

*) contrast ratio to total amount herbicides
 **) total content of herbicides in powdered iron-based mixture
 ***) content in powdered iron-based mixture

Note: Compare to Table 1 as to the herbicides symbols.

Grease conformant No.	Dry lubricates				Lubricants in powdered/lubricated mixtures				Contracting conditions				Grease compact			Results	
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Lubricant constant ¹ mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Dis. preheating temperature °C	Heating temperature for powdered lubricant mixtures °C	Compacting temperature °C	Ejection force MPa	Density	Appearance		Sectional density
	Constant ² mass%	Kid (melting point) °C	Constant ³ mass%	Kid (melting point) °C		Kid (melting point) °C	Constant ⁴ mass%	Kid (melting point) °C	Constant ⁵ mass%								
1	5	A(120°C)	95	A(118 to 132°C)	0.950	A(120°C) 0.825	50	A(118 to 132°C) 0.025	30	210	120	160-	17	2.40	-	good	laminar
2	5	A(118°C)	95	A(117°C)	0.360	A(120°C) 0.110	75	A(118 to 132°C) 0.050	25	210	150	160	19	2.41	good	good	laminar
3	5	B(120°C)	95	B(115°C)	0.350	A(120°C) 0.200	57	C(117°C) 0.150	43	210	155	170	18	2.41	good	good	laminar
4	10	B(120°C)	90	A(115°C)	0.150	A(115°C) 0.090	35	A(117°C) 0.100	67	110	120	150	20	2.29	good	good	laminar
5	10	E(116°C)	90	D(114°C)	0.050	A(115°C) 0.051	50	A(118 to 132°C) 0.055	50	210	145	160	20	2.45	good	good	laminar
6	10	F(120°C)	90	D(117°C)	0.200	A(115°C) 0.150	58	C(117°C) 0.050	40	200	155	150	16	2.43	good	good	laminar
7	20	F(120 to 230°C)	50	D(114°C)	0.400	A(120°C) 0.100	25	C(117°C) 0.100	75	210	145	158	11	2.40	-	good	laminar
8	20	G(120°C)	60	C(117°C)	0.150	A(120°C) 0.100	67	A(117°C) 0.025	33	200	140	155	11	2.46	good	good	laminar
9	20	G(120°C)	50	A(117°C)	0.050	A(120°C) 0.020	40	A(117°C) 0.015	60	200	135	150	14	2.43	good	good	laminar
10	25	G(120°C)	75	A(118 to 115°C)	0.200	A(120°C) 0.100	50	A(118 to 132°C) 0.050	50	210	150	160	11	2.42	good	good	laminar

Table 1-2

Green compact No.	Die lubricants				Lubricant weight* mass%	Lubricants in powdered iron-based mixtures				Compacting conditions				Green compacts			Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature			Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature °C	Hotting temperature for powdered iron-based mixture °C	Compacting temperature °C	Ejection force	Density	Appearance	Sectional structure	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)		Kind (melting point): Content by mass**	Content* mass%	Kind (melting point): Content by mass**	Content* mass%								
11	25	HfP-200°C	75	B1(146°C)	0.39	AQ216°C: 4.100	25	A1(141°C) 135°C: 0.110 C1(173°C): 0.100	71	200	145	12	7.41	good	good	Investigation	
13	25	HfP-200°C	75	C1(147°C)	0.19	AQ216°C: 4.100	67	A1(141°C) 135°C: 0.025 A2(177°C): 0.025	33	210	135	14	7.42	good	good	Investigation	
13	30	AQ230°C	70	A2(137°C)	0.00	AQ216°C: 4.100	25	C2(106°C): 0.100 A2(177°C): 0.100	75	180	115	12	7.40	good	good	Investigation	
14	30	AQ230°C	70	A2(137°C)	0.58	AQ216°C: 4.010 A1(141°C): 0.025 C1(177°C): 0.015	70	A2(177°C): 0.015	30	185	125	15	7.45	good	good	Investigation	
15	30	AQ230°C	70	C2(100°C)	0.08	AQ216°C: 4.100 AQ216°C: 4.100 135°C: 0.030 C1(177°C): 0.030	75	A2(177°C): 0.100	25	-185	120	13	7.42	good	good	Investigation	
16	35	AQ230°C	65	A2(137°C)	0.06	AQ216°C: 4.100 AQ216°C: 4.100	50	A2(177°C): 0.100 C1(177°C): 0.100	50	190	130	13	7.40	good	good	Investigation	
17	35	AQ216°C	65	D2(127°C)	0.50	AQ216°C: 4.025	50	A2(177°C): 0.025	50	190	120	10	7.45	good	good	Investigation	
18	35	AQ216°C	65	A1(147°C) D1(147°C)	0.08	AQ216°C: 4.130	75	A1(141°C) 135°C: 0.020	25	205	140	13	7.40	good	good	Investigation	
19	40	AQ216°C	60	B1(146°C)	0.39	AQ216°C: 4.200	57	C1(177°C): 0.100	43	200	125	17	7.47	good	good	Investigation	
20	40	AQ216°C	60	C1(147°C)	0.19	AQ216°C: 4.040	57	A2(177°C): 0.110	70	200	135	17	7.42	good	good	Investigation	

*) content ratio to total amount lubricant

**) total content of lubricant in powdered iron-based mixture

***) content in powdered iron-based mixture

Table 1-3

Gun concrete Nos.	On lubricants			Lubricant is provided from-hand mixtures			Comparative conditions			On test samples			Remarks
	Lubricant of higher melting point than comparing temperature		Kind (melting point)	Lubricant of lower melting point than comparing temperature		Kind (melting point) and comparing temperature	Dis. preheating temperature	Heating temperature for provided from-hand mixture	Comparing temperature	Ejection force	Density	Appearance	Sectional structure
	Content* mark%	Kind (melting point)	Content* mark%	Kind (melting point)	Content* mark%	Content* mark%	Content* mark%	Content* mark%	Content* mark%	Content* mark%	Content* mark%	Content* mark%	Content* mark%
21	40	AN112128 E1212128 E1212128	60	D1(146°C)	0.010	A4216C1.023	50	Al1141 155°C-0.015	155	16	7.42	good	good
22	40	AN112128 E1212128 E1212128	60	D1(137°C)	0.100	A4216C1.120	60	C1117C1.090	152	16	7.42	good	good
23	40	AN112128 E1212128 E1212128	60	D1(146°C)	0.110	A4216C1.150	43	Al1141 155°C-0.015 C1117C1.100	155	14	7.40	good	good
24	41	AN112128 E1212128 E1212128	59	A1(148 to 155°C)	0.120	A4216C1.180	67	Al1141 155°C-0.015 C1117C1.100	160	17	7.44	good	good
25	50	AN112128 E1212128 E1212128	50	A1(177°C)	0.050	A4216C1.020	48	C1117C1.015 A4217C1.015	160	16	7.43	good	good
26	50	AN112128 E1212128 E1212128	50	B1(146°C)	0.100	A4216C1.100	38	Al1141 155°C-0.015 A4217C1.090 C1117C1.015	165	19	7.41	good	good
27	50	AN112128 E1212128 E1212128	50	D1(146°C)	0.130	A4216C1.100	29	Al1141 155°C-0.015 C1117C1.100	165	20	7.41	good	good
28	2	E212128	98	D2(137°C)	0.100	A4216C1.090	30	Al1141 155°C-0.015 A4217C1.015	170	19	7.46	good	good
29	2	E212128	98	D1(146°C)	0.400	A4216C1.100	25	C1117C1.020 A4217C1.100	170	17	7.39	good	good
30	10	C30128	90	A1(148 to 155°C)	-	-	-	-	160	55	7.31	good	Comparative Example

* Content ratio to total content lubricant
 ** Content ratio to total content lubricant
 *** Content is provided from-hand mixtures

Table 1-4

Green compact No.	Die lubricants				Lubricants in powdered form-based mixtures				Compressive conditions				Green compacts			Remarks		
	Lubricant of higher melting point than compaction temperature		Lubricant of lower melting point than compaction temperature		Lubricant amount, mass%	Lubricant of higher melting point than compaction temperature		Lubricant of lower melting point than compaction temperature		Die preheating temperature, °C	Heating temperature for powdered form-based mixtures, °C	Compacting temperature, °C	Ejection force	Density	Appearance		Sectional structure	
	Content, mass%	Kind (melting point)	Content, mass%	Kind (melting point)		Kind (melting point)	Content, mass%	Kind (melting point)	Content, mass%									
31	10	A1210C3	90	B1(140°C)	0.600	A1210C3, 0.300	50	A1210C3, 0.300	50	190	123	140	29	7.33	marred	good	Comparative Example	
32	2	A1210C3	98	C1(147°C)	0.150	A1210C3, 0.150	95	C1(147°C), 0.075	5	199	123	140	31	7.34	marred	good	Comparative Example	
33	75	A1210C3	25	A1(127°C)	0.150	A1210C3, 0.090	20	C1(147°C), 0.150	80	110	115	130	17	7.40	good	void	Comparative Example	
34	0	-	100	A1(118 to 133°C)	0.350	A1210C3, 0.350	40	A1(118 to 133°C), 0.070	20	210	150	160	35	7.42	marred	good	Comparative Example	
35	100	A1210C3, 0.150 to 133°C	0	-	0.200	A1210C3, 0.100	50	A1(117°C), 0.100	50	100	125	140	38	7.37	good	good	Comparative Example	
36	100	A1210C3, 0.150 to 133°C	0	-	0.300	A1210C3, 0.225	75	A1(118 to 133°C), 0.075	25	100	50	60	35	7.27	good	good	Comparative Example	
37	0	-	100	A1(118 to 133°C) A1(118 to 133°C)	0.300	A1210C3, 0.225	75	A1(118 to 133°C), 0.075	25	270	210	220	29	7.43	voided	good	Comparative Example	
38	-	-	-	-	0.600	A1210C3, 0.150	70	A1(118 to 133°C), 0.075	30	220	160	170	38	7.35	marred	good	Comparative Example	

*1) content ratio in total amount lubricant
 *2) total content of lubricant in powdered form-based mixtures
 *3) content in powdered form-based mixtures

Table 2

Symbol	Kind of lubricants		Symbol	Kind of lubricants	
A1	Calcium stearate	Metallic soap	E1	Polyureas	Thermoplastic resin
A2	Zinc stearate		E2	Polyamide (nylon 66)	
A3	Lithium stearate		E3	Polytetrafluoroethylene	
A4	Lithium hydroxystearate		F1	Polyethylene elastomer	Thermoplastic elastomer
B1	Straight-chain low-density polyethylene		F2	Polyimide elastomer	
C1	Ethylene bis-stearamide	Acid wax	G1	Graphite	Isopropyl lamellar lubricant
C2	Monostearyl stearate		G2	MoS ₂	
D1	Ethylene bis-stearamide-polyethylene sulfonate	Eutectic mixture	G3	Carbon fluoride	
D2	Ethylene bis-stearamide-zinc stearate sulfonate		H1	Melamine cyanurate acid adduct (MCA)	Organic lamellar lubricant
D3	Ethylene bis-stearamide-calcium stearate sulfonate		E2	N-allyl sebacate- δ -allyl ester	

Table 3

Steady Compress No.	Die lubricants				Kind of free-hand powder	Quality number in free-hand pencil lubrication	Lubricants in powder free-hand lubrication						Comparing conditions				Steady Compress		Remarks
	Lubricant of higher melting point than comparing temperatures		Lubricant of lower melting point than comparing temperatures				Lubricant of higher melting point than comparing temperatures	Lubricant of lower melting point than comparing temperatures	Die preheating temperature	Heating temperature of powder free-hand lubrication	Comparing temperature	Density	Tensile strength						
	Compos. ^a mass%	Kind (melting point)	Compos. ^a mass%	Kind (melting point)															
														Compos. ^a mass%	Kind (melting point): Composed by mass%	Compos. ^a mass%	Kind (melting point): Composed by mass%		
2-1	75	Al(104) to 195°C	75	Al(104) to 195°C	0.20	Al(104) to 195°C	25	210	130	160	7.42	7.40	320					Invention	
2-2	-	-	-	-	0.6	a	Al(104) to 195°C	75	Al(104) to 195°C	25	210	130	160	7.37	7.31	740	Conventional Example		
2-3	75	Al(104) to 195°C	75	Al(104) to 195°C	0.6	b	Al(104) to 195°C	75	Al(104) to 195°C	25	210	130	160	7.42	7.43	710	Invention		
2-4	-	-	-	-	0.6	b	Al(104) to 195°C	75	Al(104) to 195°C	25	210	130	160	7.37	7.34	640	Conventional Example		
2-5	75	Al(104) to 195°C	25	Al(104) to 195°C	0.9	a	Al(104) to 195°C	20	Al(104) to 195°C	30	165	120	135	7.23	7.22	810	Invention		
2-6	-	-	-	-	0.9	a	Al(104) to 195°C	20	Al(104) to 195°C	30	165	120	135	7.17	7.12	770	Conventional Example		
2-7	75	Al(104) to 195°C	75	Al(104) to 195°C	0.9	d	Al(104) to 195°C	20	Al(104) to 195°C	30	165	120	135	7.23	7.22	830	Invention		
2-8	-	-	-	-	0.9	d	Al(104) to 195°C	20	Al(104) to 195°C	30	165	120	135	7.25	7.23	760	Conventional Example		
2-9	75	Al(104) to 195°C	25	Al(104) to 195°C	0.1	e	Al(104) to 195°C	20	Al(104) to 195°C	30	170	115	130	7.36	7.25	620	Invention		
2-10	-	-	-	-	0.1	e	Al(104) to 195°C	20	Al(104) to 195°C	30	170	115	130	7.27	7.14	510	Conventional Example		
2-11	75	Al(104) to 195°C	25	Al(104) to 195°C	0.1	f	Al(104) to 195°C	20	Al(104) to 195°C	30	170	115	130	7.25	7.14	630	Invention		
2-12	-	-	-	-	0.1	f	Al(104) to 195°C	20	Al(104) to 195°C	30	170	115	130	7.16	7.05	590	Conventional Example		
2-13	75	Al(104) to 195°C	25	Al(104) to 195°C	0.2	a	Al(104) to 195°C	20	Al(104) to 195°C	30	210	130	160	7.40	7.39	820	Invention		

Sintered Compact Nos.	Die lubricants				Kind of iron-based powder provides	Graphite content in iron-based powder lubricant, mass%	Lubricants in powdered iron-based sinter						Compacting conditions			Over compression	Sintered Compacts		Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature				Lubricant content, mass%	Lubricant of higher melting point than compacting temperature	Lubricant of lower melting point than compacting temperature	Die preheating temperature, °C	Holding temperature for powdered iron-based sinter, °C	Compacting temperature, °C	Density	Tensile strength					
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)															
															Content* mass%		Kind (melting point)	Content* mass%	
2-14	75	AJ200°C	25	AJ148 to 155°C	0	1.1	0.10	AJ200°C x 15	75	AJ148 to 155°C x 10	210	150	160	7.36		750			MPa
2-15	75	AJ200°C	25	AJ148 to 155°C	0	1.3	0.10	AJ200°C x 15	75	AJ148 to 155°C x 10	210	150	160	7.23	310		breakdown		

* Content ratio to total amount lubricant
 -) Kind of lubricant in powdered iron-based sinter
 -) Content of graphite in iron-based sinter
 Note: Content in Table 2 is in the lubricant symbols.

Example 3

[0075] A partially alloyed steel powder of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder was used as an iron-based powder. Iron-based powder mixtures were prepared by mixing this alloyed steel powder with 0.2% by mass of a graphite powder and various lubricants shown in Table 3. The mixing was effected with heat and by use of a high-speed mixer.

[0076] First, a pressure compaction die was preheated at each of the temperatures listed in Table 4. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the die surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 4. The temperature measured on the die surface was taken as a pressure compaction temperature.

[0077] Subsequently, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 4. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 4.

[0078] As a conventional example, a similar rectangular green compact (Green compact No. 38) was formed in the same manner as in Example 4 except that a die was not coated with a die lubricant for warm compaction with die.

[0079] After completion of the compaction, the ejection force was measured.

[0080] With regard to each of the resultant green compacts, the density was determined by Archimedes' principle. Visual inspection was then made of the appearance of the green compact to find faults such as marring, breakage and the like. The green compact was centrally cut, embedded in resin and then abraded, followed by examination of voiding in section on a light microscope.

[0081] The drawing force, density, appearance and sectional structure of the green compact are tabulated in Table 4.

[0082] All the green compacts according to this invention show as low a drawing force after compaction as 20 MPa or below and as high a density as 7.43 Mg/m³ or above. In addition, each such product causes neither surface oxidation resulting from heating nor faults such as marring, breakage and the like. The sectional structure is normal with the absence of coarse voids.

[0083] The comparative and conventional examples that depart from the scope of the invention suffered a high drawing force exceeding 20 MPa, a low density of less than 7.39 Mg/m³, or coarse voids near to the sectional surface of the green compact.

[0084] The present invention is highly advantageous in that a high-density green compact is obtainable with superior appearance and sectional structure as well as low drawing force.

Table 4-1

Grease compact No.	Die lubricants				Lubricants by synthetic free-based esters				Comparing conditions				Grease compact			Remarks
	Lubricant of higher melting point than comparing temperature		Lubricant of lower melting point than comparing temperature		Lubricant of higher melting point than comparing temperature		Lubricant of lower melting point than comparing temperature		Die preheating temperature	Heating temperature for powdered free-based mixture	Comparing temperature	Ejection force	Density	Appearance	Sectional structure	
	Content* mass%	K.L.M (melting point)	Content* mass%	K.L.M (melting point)	Kind (melting point) Content by mass%	Content* mass%	Kind (melting point) Content by mass%	Content* mass%								
1	2	A1(230°C)	94	A1(148 to 155°C)	94	A1(230°C) 0.035	20	A1(148 to 155°C) 0.035	30	190	150	17	7.44	good	good	Injection
2	3	A2(180°C)	92	A2(127°C)	87	A1(230°C) 0.100	67	A1(148 to 155°C) 0.019	33	190	150	18	7.43	good	good	Injection
3	5	B1(140°C)	91	B1(140°C)	87	A1(230°C) 0.200	37	C1(177°C) 0.150	41	180	140	17	7.44	good	good	Injection
4	10	B2(160°C)	90	A2(127°C)	87	A2(160°C) 0.050	31	A2(177°C) 0.100	67	160	120	20	7.43	good	good	Injection
5	10	B3(140°C)	90	D1(117°C)	80	A2(160°C) 0.025	30	A1(148 to 155°C) 0.035	30	190	145	20	7.43	good	good	Injection
6	15	F1(200°C)	85	D2(127°C)	80	A2(160°C) 0.120	69	C1(177°C) 0.080	40	180	135	19	7.47	good	good	Injection
7	20	F2(200 to 210°C)	80	D1(117°C)	25	A1(230°C) 0.100	25	C1(177°C) 0.150	75	190	145	11	7.43	good	good	Injection
8	10	D1(200°C)	90	C1(147°C)	87	A1(230°C) 0.100	67	A2(177°C) 0.025	33	185	140	11	7.44	good	good	Injection
9	20	D2(200°C)	80	A2(127°C)	40	A1(230°C) 0.060	40	A2(177°C) 0.060	60	180	135	14	7.47	good	good	Injection
10	20	D1(200°C)	80	A1(148 to 155°C)	30	A1(230°C) 0.100	30	A1(148 to 155°C) 0.035 A2(177°C) 0.030 C1(177°C) 0.025	50	190	150	12	7.45	good	good	Injection

* values refer to total amount lubricant
 ** total content of lubricant is petroleum free-based esters
 *** content in petroleum free-based esters
 Note: Content refers to Table 2 in the Japanese original.

Table 4-2

Gears compact Nos.	Die lubricants				Lubricants in production iron-based steels				Compacting coefficient			Open compacts			Remarks		
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Lubricant weight* mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature °C	Molding temperature for powder of iron-based steels °C	Compacting temperature °C	Ejection force MPa	Density		Appearance	Radical structure
	Content* mass%	Kind (melting point) °C	Content* mass%	Kind (melting point) °C		Content* mass%	Kind (melting point) °C	Content* mass%	Kind (melting point) °C								
11	25	H10-200°C	75	B1(145°C)	0.308	A4(16°C) 0.106	50	A1(14 to 15°C) 0.060 C1(17°C) 0.040	50	190	143	138	14	7.45	good	good	Ironation
12	50	H10-200°C	50	C1(145°C)	0.118	A4(16°C) 0.106	67	A1(14 to 15°C) 0.033 A1(17°C) 0.013	33	190	155	160	13	7.46	good	good	Ironation
13	30	A4(16°C)	70	A1(17°C)	0.009	A4(16°C) 0.106	25	C1(16°C) 0.200 A1(17°C) 0.100	75	160	120	130	12	7.45	good	good	Ironation
14	60	A4(16°C)	40	A1(17°C)	0.110	A4(16°C) 0.050 A1(16°C) 0.011 C1(17°C) 0.041	67	A1(17°C) 0.045	33	165	125	135	14	7.46	good	good	Ironation
15	30	A4(16°C)	70	C1(180°C)	0.009	A4(16°C) 0.106 A4(16°C) 0.106 A1(14 to 15°C) 0.050 C1(17°C) 0.015	75	A1(17°C) 0.100	25	160	120	130	13	7.46	good	good	Ironation
16	25	A4(16°C)	65	A1(17°C)	0.308	A4(16°C) 0.094 A4(16°C) 0.050	50	A1(17°C) 0.050 C1(16°C) 0.050	50	170	120	140	15	7.45	good	good	Ironation
17	25	A4(16°C)	65	D1(127°C)	0.118	A4(16°C) 0.117	17	A1(17°C) 0.013	17	170	135	150	16	7.47	good	good	Ironation
18	35	A4(16°C)	65	A1(16°C) 35 D1(17°C) 50	0.308	A4(16°C) 0.116	75	A1(14 to 15°C) 0.030	25	190	140	155	14	7.44	good	good	Ironation
19	60	A4(16°C)	40	B1(145°C)	0.318	A4(16°C) 0.200	17	C1(17°C) 0.100	43	160	135	170	17	7.46	good	good	Ironation
20	40	A4(16°C) 30	60	C1(145°C)	0.118	A4(16°C) 0.040	17	A1(17°C) 0.110	73	160	125	150	17	7.46	good	good	Ironation

* content ratio in steel compact lubricant
 ** total content of lubricant in production iron-based steels
 *** content in production iron-based steels

Table 4-3

Oxon Control No.	Die lubricant				Lubricant in powdered free-based esters				Compression conditions				Gaseous products			Results
	Lubricant of higher melting point than compressing temperature		Lubricant of lower melting point than compressing temperature		Lubricant content** mark%	Lubricant of higher melting point than compressing temperature		Lubricant of lower melting point than compressing temperature		Die preheating temperature °C	Heating temperature for powdered free-based esters °C	Compressing temperature °C	Density	Appearance	Solidified structure	
	Content* mark%	Kind (melting point)	Content* mark%	Kind (melting point)		Content* mark%	Kind (melting point)	Content* mark%	Kind (melting point)							
31	40	AQ116CQ30 E226CQ30	60	D1(414°C)	0.150	AQ16CQ3.100	47	AI(14) = 15°CQ3.050	77	190	140	135	7.46	good	good	Inversion
32	40	AQ116CQ30 E226CQ30	60	D3(413°C)	0.300	AQ16CQ3.100	48	CH14CQ3.120	68	180	135	130	7.45	good	good	Inversion
33	50	FI005CQ31 AQ115CQ35	50	D1(414°C)	0.350	AQ28CQ3.100	45	AI(14) = 15°CQ3.100 CH14CQ3.100	37	190	140	135	7.44	good	good	Inversion
34	50	E226CQ31 E226CQ32	50	AI(14) = 15°C	0.150	AQ28CQ3.100	47	AI(14) = 15°CQ3.05 AI(12)CQ3.015	33	190	136	140	7.48	good	good	Inversion
35	60	E226CQ30 FI0220CQ30	40	AQ17CQ	0.300	AQ28CQ3.020	40	CH14CQ3.015 AI(12)CQ3.015	46	180	140	150	7.47	good	good	Inversion
36	70	AQ28CQ31 E226CQ31	30	B1(146°C)	0.200	AQ28CQ3.100	30	AI(14) = 15°CQ3.051 AI(17)CQ3.050 CH14CQ3.051	56	190	155	145	7.44	good	good	Inversion
37	80	AQ116CQ30 E226CQ30 FI005CQ30	20	D1(414°C)	0.350	AI(16)CQ3.100	29	AI(14) = 15°CQ3.100 CH14CQ3.100	71	190	155	145	7.45	good	good	Inversion
38	2	E226CQ3	98	D3(413°C)	0.300	AQ16CQ3.100	50	AI(14) = 15°CQ3.050 AI(12)CQ3.015	50	200	160	170	7.49	good	good	Inversion
39	3	E226CQ3	97	D3(414°C)	0.400	AQ16CQ3.100	23	CH14CQ3.050 AI(12)CQ3.015	75	200	160	170	7.43	good	good	Inversion
30	10	AQ28CQ	90	AI(148) =	-	-	-	-	-	190	145	140	7.33	good	good	Compressive Example

*) content ratio to total solvent lubricant
 **) total content of lubricant in powdered free-based esters
 ***) content in powdered free-based esters

Table 4-4

Green Component No.	Die lubricants				Lubricants in powdered form-based mixtures				Compressing conditions				Green compacts				Remarks
	Lubricant of higher melting point and corresponding temperature		Lubricant of lower melting point and corresponding temperature		Lubricant content by mass%	Lubricant of higher melting point and corresponding temperature		Lubricant of lower melting point and corresponding temperature		Die preheating temperature	Heating temperature of powder and lubricant mixture	Compressing temperature	Ejection time	Density	Appearance	Sectional structure	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)		Kind (melting point): Content by mass%	Content* mass%										
31	10	A323PC	90	B114PC	0.600	A323PC:0.300	50	A323PC:0.100	50	180	125	150	20	7.36	marred	good	Comparative Example
32	5	A323PC	95	C114PC	0.150	A323PC:0.145	95	C114PC:0.005	2	180	125	110	31	7.39	marred	good	Comparative Example
33	98	A323PC	10	A323PC	0.150	A323PC:0.070	20	C110PC:0.130	80	160	115	110	21	7.42	good	void	Comparative Example
34	9	-	100	A1148 to 155°C	0.300	A323PC:0.200	67	A1148 to 155°C:0.010	55	180	150	160	25	7.44	marred	good	Comparative Example
35	100	A323PC:90 A1148 to 155°C	0	-	0.200	A323PC:0.180	50	A323PC:0.100	50	170	125	140	30	7.31	good	good	Comparative Example
36	100	A323PC:15 A1148 to 155°C	0	-	0.300	A323PC:0.225 A1148 to 155°C:0.075	100	-	5	100	60	70	25	7.31	good	good	Comparative Example
37	8	-	100	A1148 to 155°C A323PC:95	0.300	A323PC:0.225	70	A1148 to 155°C:0.075	25	250	210	210	50	7.47	oddend	good	Comparative Example
38	-	-	-	-	0.600	A323PC:0.410	70	A1148 to 155°C:0.180	30	200	160	170	38	7.39	marred	good	Comparative Example

*) content ratio to total content lubricant
 **) total content of lubricant in powdered form-based mixture
 ***) content in powdered form-based mixture

Example 4

[0085] The following two different powders were used as iron-based powders; namely (1) a partially alloyed steel powder a of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder, and (2) a prealloyed steel powder b of a Fe-3Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V.

[0086] The partially alloyed steel powder a, and prealloyed steel powder b were mixed with graphite in the contents shown in Table 5 and the lubricants shown in Table 5, whereby iron-based powder mixtures were prepared. The mixing was effected with heat and by use of a high-speed mixer. The content of graphite is by a mass ratio relative to the total amount of the iron-based powder mixture.

[0087] First, a die was preheated at each of the temperatures listed in Table 5. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the die surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 5. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0088] Secondly, the die thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 5.

[0089] A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 5.

[0090] With regard to each iron-based green compact thus obtained, the density was determined by Archimedes' principle as in Example 1.

[0091] Subsequently, the iron-based green compact was sintered in a N₂-10%H₂ atmosphere and at 1,130 C for 20 minutes, whereby an iron-based sintered compact was formed. The density of the resultant sintered compact was determined by Archimedes' principle. The test results are tabulated in Table 5. The examples of the invention provides high densities.

[0092] As stated above, the present invention is significantly advantageous in that a high-density green compact can be produced with superior appearance and sectional structure and by single compaction. Drawing of the product from the associated mold is possible at a low force with a prolonged lifetime of the die. Also notably, a high-density sintered compact is easy to produce.

Table 5

Sintered Compact No.	Die lubricants			Kind of free-based powder	Oxide content in free-based powder, mass%	Lubricants in powdered free-based lubricants					Compacting conditions			Green compact Density	Sintered compact Density	Remarks
						Lubricant amount, mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature °C	Harding temperature for powdered free-based lubricant °C	Compacting temperature °C			
	Content* mass%	Kind (melting point)	Content by mass%				Kind (melting point) Content by mass%									
								Content* mass%	Kind (melting point)	Content by mass%				Kind (melting point) Content by mass%		
2-1	75	AJ(200°C)	25	Al(148 to 155°C)	0.15	AJ(200°C):0.1	67	Al(148 to 155°C):0.025 AJ(177°C):0.075	31	190	150	160	7.49	7.47	Invention	
2-2	75	AJ(200°C)	25	Al(148 to 155°C)	0.30	AJ(200°C):0.1	67	Al(148 to 155°C):0.025 AJ(177°C):0.075	31	190	150	160	7.47	7.43	Invention	
2-3	75	AJ(200°C)	25	Al(148 to 155°C)	0.45	AJ(200°C):0.1	67	Al(148 to 155°C):0.025 AJ(177°C):0.075	31	190	150	160	7.45	7.43	Invention	
2-4	75	AJ(200°C)	25	Al(148 to 155°C)	0.55	AJ(200°C):0.1	67	Al(148 to 155°C):0.025 AJ(177°C):0.075	31	190	150	160	7.39	7.38	Comparative Example	
2-5	75	AJ(200°C)	25	AJ(177°C)	0.15		50	Al(148 to 155°C):0.05 C(147°C):0.05	50	165	120	155	7.34	7.24	Invention	
2-6	75	AJ(200°C)	25	AJ(177°C)	0.20		50	Al(148 to 155°C):0.05 C(147°C):0.05	50	165	120	155	7.31	7.22	Invention	
2-7	75	AJ(200°C)	25	AJ(177°C)	0.45		50	Al(148 to 155°C):0.05 C(147°C):0.05	50	165	120	155	7.30	7.29	Invention	
2-8	75	AJ(200°C)	25	AJ(177°C)	0.55		50	Al(148 to 155°C):0.05 C(147°C):0.05	50	165	120	155	7.25	7.24	Comparative Example	

*1) content ratio of solid anhydrous lubricant
*2) total content of lubricant in powdered free-based lubricant
*3) content in powdered free-based lubricant
Note: Content ratio in Table 3 is in the lubricant symbols.

Claims

1. A die lubricant for warm compaction with die lubrication, comprising a mixture of a lubricant having a higher melting point than a preset compaction temperature, and a lower melting point than the compaction temperature, the die lubricant for warm compaction with die lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted by pressure compaction.
2. A die lubricant for warm compaction with die lubrication, comprising a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lower melting point than the compaction temperature and as the balance, the die lubricant for warm compaction with die lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted by pressure compaction.
3. The die lubricant for warm compaction with die lubrication according to claim 2, wherein the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.
4. The die lubricant for warm compaction with die lubrication according to claim 2, wherein the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.
5. An iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder and a powder compaction lubricant, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.
6. An iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.
7. The iron-based powder mixture for warm compaction with die lubrication, wherein the content of the powder compaction lubricant is in the range from 0.05 to 0.40% by mass.
8. A process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a die lubricant for warm compaction with die lubrication to a surface of the die by means of electrical charging; filling a heated iron-based powder mixture in the die; and then subjecting the powder mixture to pressure compaction at a preset compaction temperature, wherein the lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder and a powder compaction lubricant, the powder compaction lubricant comprising a lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.
9. A process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a lubricant for warm compaction with die lubrication to the surface of the die by means of electrical charging; filling a heated iron-based powder mixture into the die; and then subjecting the powder mixture to pressure compaction at a present compaction temperature, wherein the die lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder, a powder compaction lubricant and a graphite powder, the powder compaction lubricant comprising a lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to

75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance; and a graphite powder being added in a content less than 0.5% by mass based on the total amount of the iron-based powder mixture.

- 5 10. The process according to one of claims 9 and 10, wherein the higher-melting die lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.
- 10 11. The process according to one of claims 8 and 9, wherein the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.
- 15 12. The process according to any one of claims 8 to 11, wherein the lubricant for warm compaction lubrication is added in a content from 0.05 to 0.40% by mass.
- 20 13. A process for the production of a high-density iron-based sintered compact, comprising the step of sintering the high-density iron-based green compact produced by the process according to any one of claims 8 to 12, thereby forming a sintered compact.
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INTERNATIONAL SEARCH REPORT

International application No.

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A. CLASSIFICATION OF SUBJECT MATTER Int.Cl ⁷ B22F 1/00, 3/02, 3/035, C22C 33/02		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) Int.Cl ⁷ B22F 1/00, 3/02, 3/035, C22C 33/02		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Jitsuyo Shinan Koho 1926-1996 Toroku Jitsuyo Shinan Koho 1994-2000 Kokai Jitsuyo Shinan Koho 1971-2000 Jitsuyo Shinan Toroku Koho 1996-2000		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP, 10-280005, A (Kawasaki Steel Corporation), 20 October, 1998 (20.10.98) (Family: none)	1-13
Y	JP, 11-193404, A (Hitachi Powdered Metals co., Ltd.), 21 July, 1999 (21.07.99) (Family: none)	1-13
Y	US, 5744433, A (Storstrom et al.), 28 April, 1998 (28.04.98) & JP, 10-501270, A (Hoganasu Akuchiboragetto), 03 February, 1998 (03.02.98)	1-13
Y	JP, 9-71801, A (Kawasaki Steel Corporation), 18 March, 1997 (18.03.97) (Family: none)	1-13
Y	US, 5682591, A (Inculet et al.), 28 October, 1997 (28.10.97) & JP, 8-100203, A	1-13
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Date of the actual completion of the international search 24 October, 2000 (24.10.00)		Date of mailing of the international search report 07 November, 2000 (07.22.00)
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